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FIRST ANNUAL PROGRESS REPORT ON A STUDY OF CORROSION IN MUNICIPAL INCINERATORS

BATTELLE COLUMBUS LABORATORIES

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ON A STUDY OF CORROSION IN MUNICIPAL INCINERATORS

By

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The objective of this research was to obtain a better understanding of factors that contribute to the corrosion of structural and energy recovery components in systems designed to incinerate municipal refuse. Specifically, the corrosion problems associated with the burning refuse containing high concentrations of polyvinyl chloride were evaluated for a variety of alloys having potential use in water-wall heat-recovery systems and in air pollution control devices. The effect of municipal refuse composition, particularly the PVC content, on the corrosion and metal wastage of alloys having potential use in fireside heat recovery systems and air pollution devices was investigated by inserting corrosion probes in the Miami County, Ohio Incinerator.

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FOREWORD

Man and his environment must be protected from the adverse effects of pesticides, radiation, noise and other forms of pollution, and the unwise management of solid waste. Efforts to protect the environment require a focus that recognizes the interplay between the components of our physical environment--air, water, and land. The National Environmental Research Centers provide this multidisciplinary focus through programs engaged in

- studies on the effects of environmental contaminants on man and the biosphere, and
- a search for ways to prevent contamination and to recycle valuable resources.

In an attempt to solve the problem of refuse disposal, the study described here utilized corrosion probes that were inserted in the Miami County Ohio Incinerator. The effect of the polyvinyl chloride content on the corrosion and metal wastage of alloys of potential use in fireside heat recovery systems and air pollution devices was investigated.

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ABSTRACT

The effect of municipal refuse composition, particularly the PVC content, on the corrosion and metal wastage of alloys of potential use in fireside heat recovery systems and air pollution devices was investigated by inserting corrosion probes in the Miami County Ohio Incinerator. The results show that the corrosion rate of low-alloy steel (A106 and A213-T11) is markedly increased by PVC content and metal temperature while the corrosion rates of stainless steels (Types 310, 316, 321, and 446) or Incoloy 825 are not affected by these variables. An increase in the PVC content by 0.5 percent is sufficient to produce a noticeable increase in the corrosion rate of low-alloy steels while as much as 2 percent PVC has little or no effect upon the rate of attack of the stainless steels. Increasing the incinerator gas temperature, however, from 1400 F to 1550 F increases the corrosion rates of both the low- and high-alloy steels by as much as 6X for specimen temperatures above 800 F.

Studies of the deposits and the reaction products reveal that for metal temperature between 400 F and 800 F, the attack is by a chloride reaction while at temperatures above 800 F, the attack is one of a chloride assisted sulfide reaction. Increasing the PVC content of the refuse increases the attack of the low-alloy steels in the low (400-800 F) temperature range by increasing the continuity of the ferrous chloride layer at metal surface. At higher temperatures, the major attack is by sulfidization which is assisted by chloride destruction of the semiprotective oxide layer.

The amount of inorganic chloride formed and retained by the deposit on the corrosion probe increases with the PVC content of the refuse but decreases rapidly with increasing metal temperature or with increasing gas temperature.

A STUDY OF CORROSION IN MUNICIPAL INCINERATORS

by

D. A. Vaughan, P. D. Miller, W. K. Boyd,
H. H. Krause, and P. R. Webb

OBJECTIVES

The objective of this research is to obtain a better understanding of factors that contribute to the corrosion of structural and energy recovery components in systems designed to incinerate municipal refuse. Specifically, the corrosion problems associated with the burning refuse containing high concentrations of polyvinyl chloride were evaluated for a variety of alloys having potential use in water-wall heat-recovery systems and in air pollution control devices.

INTRODUCTION

As it is becoming evident that more and more attention is being given to refuse as an energy source, the disposal of refuse by incineration can become more economical providing this source of fuel does not create pollution problems. Most of the pollution-control devices require that heat (energy) be extracted to permit operation of the flue-gas cleaning systems. This can be achieved by water-wall tubes and convection pass tubes inserted in the hot gases to absorb the heat. The technology of water-wall incinerators has advanced to a state of rather extensive use in Europe and several are operating in the United States. Corrosion problems have been reported, however, in some of the operating units. Municipal refuse is also being considered as a supplementary fuel to existing fossil fuel power generating stations and for industrial needs in process steam. To provide guidance in materials selection for both pollution control and heat recovery systems, the magnitude of the corrosive attack by the hot gases and deposits have been conducted in a conventional ceramic-lined incinerator operating with municipal refuse.

Under a previous research grant (EP00325) supported by the Solid Waste Management Office of EPA, Battelle investigated the mechanism and extent of fireside metal wastage by incineration of municipal refuse. On the basis of this study, corrosion mechanisms were proposed to explain the attack occurring over a range of metal temperatures. Furthermore, through a supplement to this grant, materials were evaluated for construction of wet-scrubber-air-pollution devices. These results have been published in "Corrosion Studies in Municipal Incinerators", (SW-72-3-3), by the National

Environmental Research Center, Cincinnati, Ohio, and in parts in various technical journals. Based upon these previous investigations, it was shown that chlorine, as HCl and/or solid chloride deposits, contribute significantly to high- and low-temperature corrosion of structural material in incinerators.

A small ($< 5\%$ of total) but increasing quantity of municipal refuse is defined as plastics which present specific problems with respect to: (1) the burning; (2) the corrosive character of the combustion products; and (3) the environmental hazard of the emitted combustion gases from the incineration process. Although burning of plastics in well designed incinerators at normal operating temperatures (1700-2000 F) with an excess of air would not be expected to present difficulties, slight grate damage has been observed. In the burning of PVC, which accounts for $\sim$ 30 percent of the plastic production, the chloride content of the combustion gases will increase. Other plastic can be expected to contribute other corrosive, toxic, or obnoxious gases.

As the mount of plastic (> 30% of which is polyvinyl chloride) in municipal refuse increases, it is anticipated that chloride corrosion will become a more serious problem. Heimberg[1] * observed drastic corrosion of the furnaces and transfer lines during incineration of PVC to evaluate off gas emission which is 30 to 40 percent HCl. Several investigations[1-4] have shown the HCl content of the flue gas and the chlorides concentration of fly ash to increase linearly with PVC content of the refuse. Because of the high dilution of flue gases entering the atmosphere, the amount of PVC incinerated would have to be 4 to 6 percent of the total refuse to exceed permissible air pollution standards of 0.7 mg/m^3 based upon experience in Europe. This amount of PVC is 10 to 20 times that in present-day municipal wastes. Some industrial wastes will, however, contain much higher concentrations of PVC and other chlorinated plastics. Although there are no regulations on HCl emission from the incineration of refuse, the experiences of Heimburg[1] and of Fessler, Leib, and Spahn[5], indicate that corrosion of heat exchangers can be severe with increased PVC loading of the refuse. These observations and the results of previous grant studies made at BCL emphasize the importance of chlorine on the mechanism of corrosion. The present investigation was initiated to explore the effect of refuse composition on corrosion with the first year of this program directed toward an evaluation of the PVC content.

EXPERIMENTAL PROGRAM

The experimental program consisted of (1) determining the corrosion rates for low-alloy steels and for stainless steels during exposure to the hot combustion gases of Miami County refuse to which various (0 to 2%) amounts of PVC were added; (2) analyses of the

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* References are listed on page 32.

deposits and corrosion products; (3) analysis of the incinerator gas composition; (4) determining the municipal to industrial waste ratio; and (5) analysis of grab samples for: (a) types (paper, wood, metal, etc.) of refuse, (b) gases evolved; and (c) ash composition. The results of Items 4 and 5 are appended to this report as they contain data incidental to the corrosion rate studies. The corrosion experiments, which consisted of 10 hour exposures, were designed to evaluate the effect of varying the amount of PVC added to normal refuse on the corrosion rate of metal, but were not intended to establish long term performance.

Corrosion Probe Design

The probe was designed to include 34 cylindrical specimens nested together end to end and then inserted into the incinerator through a side wall. The section of the probe extending through the wall was water cooled, while the specimens exposed within the furnace were cooled by air flowing inside the tubular specimens. A computer analysis had been used in previous programs to ascertain the geometry of the internal support tube required to give the most linear specimen temperature variation over the range of about 350 to 1100 F for a probe with 34 specimens. Each specimen was about 1.25 inches in OD, 1.00 inch in ID, and 1.5 inches long. The composition of the alloys used to make up the corrosion probes is given in Table 1.

Figure 1 is a schematic of the final exposure-probe apparatus. The specimens are nested together with lap joints as shown in Detail A, and retained axially at the cooling-air-outlet end by a retainer which is fixed to the internal support tube with webs as shown in Section A-A. The axial restraining force in the internal support tube is obtained by compressing the spring on the air-inlet end of the probe at assembly. Also, the spring compensates for differential thermal expansion between the specimens and the internal support tube.

Specimen temperatures are measured at four stations with Type K thermocouples either welded into the wall of the specimens or inserted into recesses drilled lengthwise into one end of the appropriate specimen. The thermocouple lead wires are brought out of the probe through the center of the internal support tube, so that temperatures can be recorded continuously on a strip-chart potentiometer recorder. Since the computed results indicated that the temperature variation is linear for regions with a constant gap between the internal support tube and the specimens, the four temperatures accurately define the specimen temperatures.

The specimen temperatures are controlled by regulating the amount of cooling air admitted to the probe. The output from a control thermocouple, which is attached to the specimen at the same axial location at the Thermocouple 3, is monitored by a proportional temperature controller. At the start of a test, the controller maintains this temperature by varying the amount of cooling air bypassing the probe through a motorized butterfly valve located between the blower and

TABLE 1. NOMINAL COMPOSITIONS OF STEELS USED IN THE CORROSION PROBE

	Composition of Steel, weight percent						
	A106-Grade B	A213-T11	Incoloy 825	Type 310	Type 316	Type 321	Type 446
Carbon, max	0.30	0.15	0.03	0.15	0.08	0.08	0.20
Manganese	0.29-1.06	0.30-0.60	0.5	2.0	2.0	2.0 (max)	1.5
Phosphorus, max	0.48	0.03	---	0.04	0.04	0.03	0.04
Sulfur, max	0.058	0.03	---	0.03	0.03	0.03	0.03
Silicon, min	0.10	0.50-1.0	0.25	0.75 (max)	0.75 (max)	0.75 (max)	0.75 (max)
Chromium	---	1.0-1.5	21.5	24-26	16-18	17-20	23-30
Molybdenum	---	0.44-0.65	3.0	---	2-3	---	---
Nickel	---	---	42.0	19-22	11-14	9.0-13	0.5 (max)
Titanium	---	---	---	---	---	Min 5 times carbon Max 0.60	---
Copper	---	---	2.25	---	---	---	---

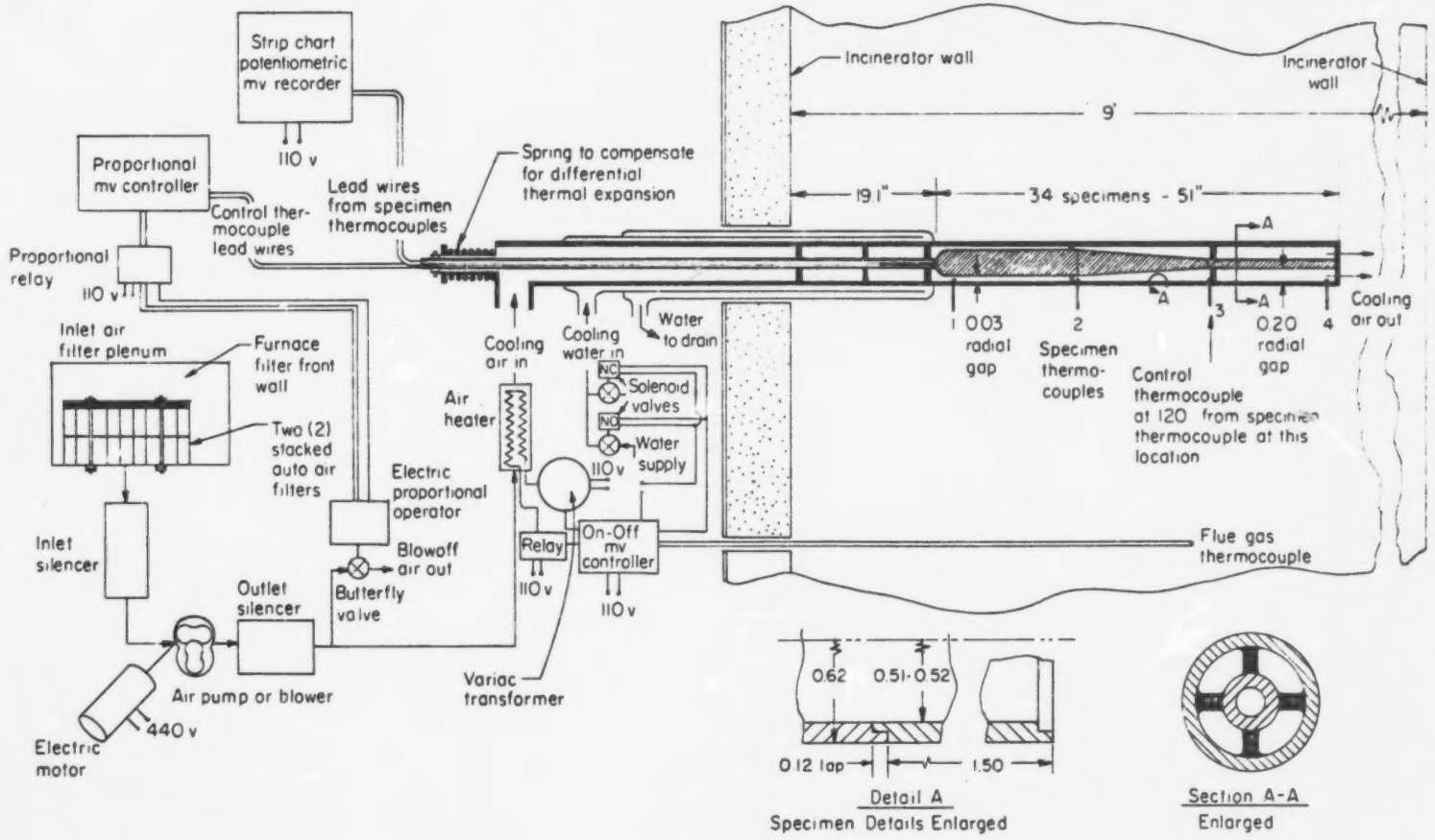

FIGURE 1. SCHEMATIC OF SPECIMEN-EXPOSURE-PROBE APPARATUS

the probe. A Roots-blower air pump delivering up to about 34 cfm was used with a 5-hp motor as a drive.

As indicated in Figure 2, the specimen temperatures varied somewhat from one experiment to another and these differences must be considered in evaluating the effect of the PVC addition on the corrosion behavior. This variation is in part a result of incinerator operation and in part the capacity of the pump used for the cooling air, the amount of which was controlled by a thermocouple on Specimen 19 of each probe. The incinerator gas temperature variation as tabulated below is determined by the type of refuse and to some extent by operator choice. Two portholes in the Miami County Incinerator, Figure 3, were employed to obtain a temperature differential of 150 F during Run 19/20.

Probe			Average Gas Temperature	
Number	PVC Addition, %	Location	Chart Record	From Probe
16	0	1	1290	
17	2	1	1310	
18	1	1	1600	
19	0.5	1	1260	1400
20	0.5	2	1260	1495 (1550 F)

The temperatures taken from the chart records of the incinerator control panel cycled by as much as 150 F above and below these averages. These temperature records were obtained from a thermocouple in the side of the combustion chamber opposite the corrosion probe insertion port and at a somewhat higher level in the furnace while the thermocouples for probe temperatures were near the end of the corrosion probes in Run 19/20. In the case of Probe 20, the thermocouple extended only one or two inches past the cooling gas exit port. The exit cooling gas is believed to have lowered this reading as a temperature profile study of various ports in the incinerator showed the temperature differential between the ports, in which Probes 19 and 20 were inserted, to be 150 F.

The temperatures of corresponding specimens for Probes 19 and 20 were held to within about 50 F, but the first 20 specimens were at as much as 200 F lower temperature than the other probes. In the case of Probe 18, the temperatures of Specimens 16-34 were significantly (80 - 100 F) higher than those for other probe runs. These higher specimen temperatures resulted from the higher gas temperatures in the incinerator for this experiment. Probes 19 and 20 were exposed simultaneously at positions having 150 F difference in gas temperature to evaluate this effect on the corrosion rate.

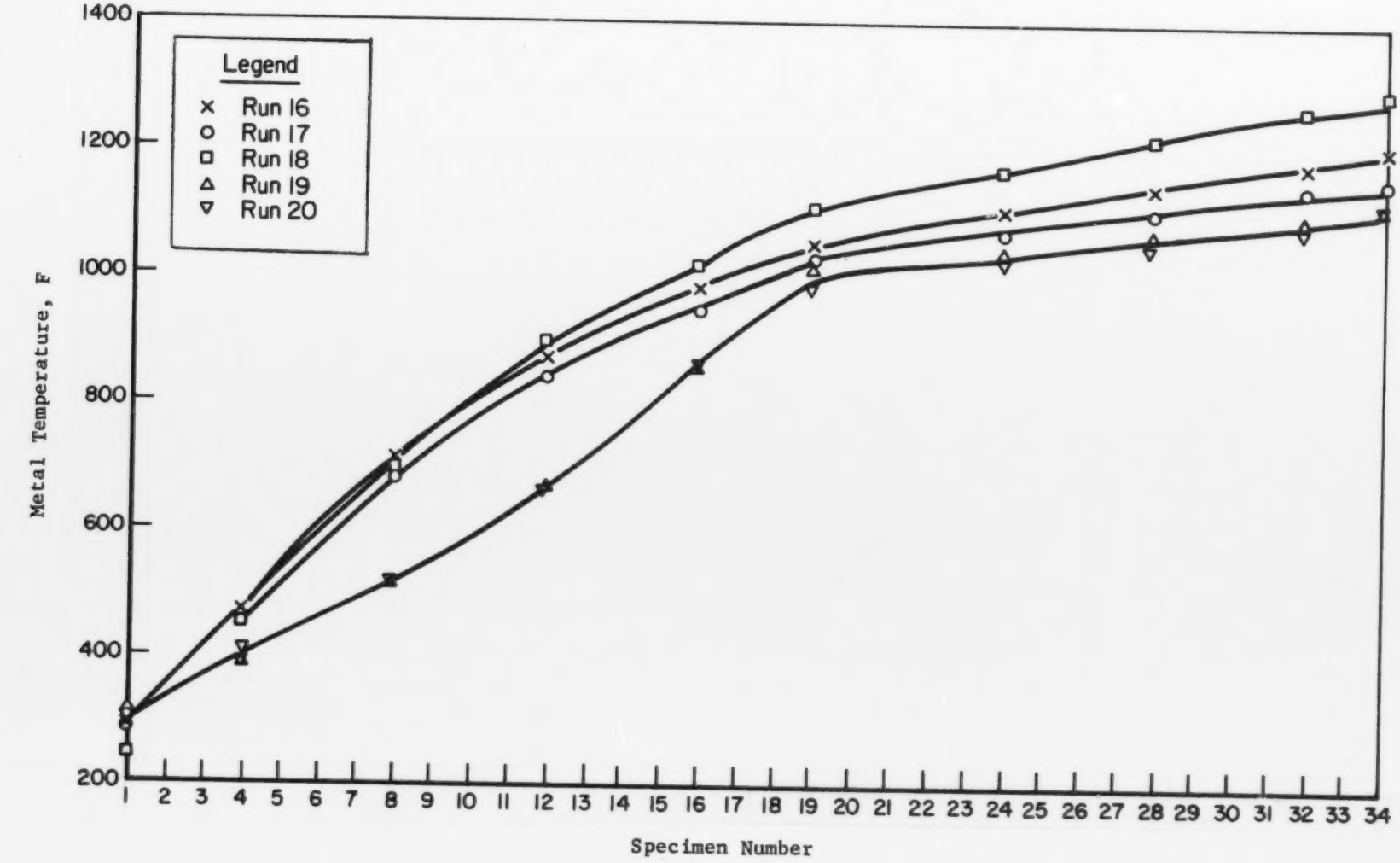

FIGURE 2. TEMPERATURE VERSUS SPECIMEN NUMBER FOR FIVE EXPERIMENTS

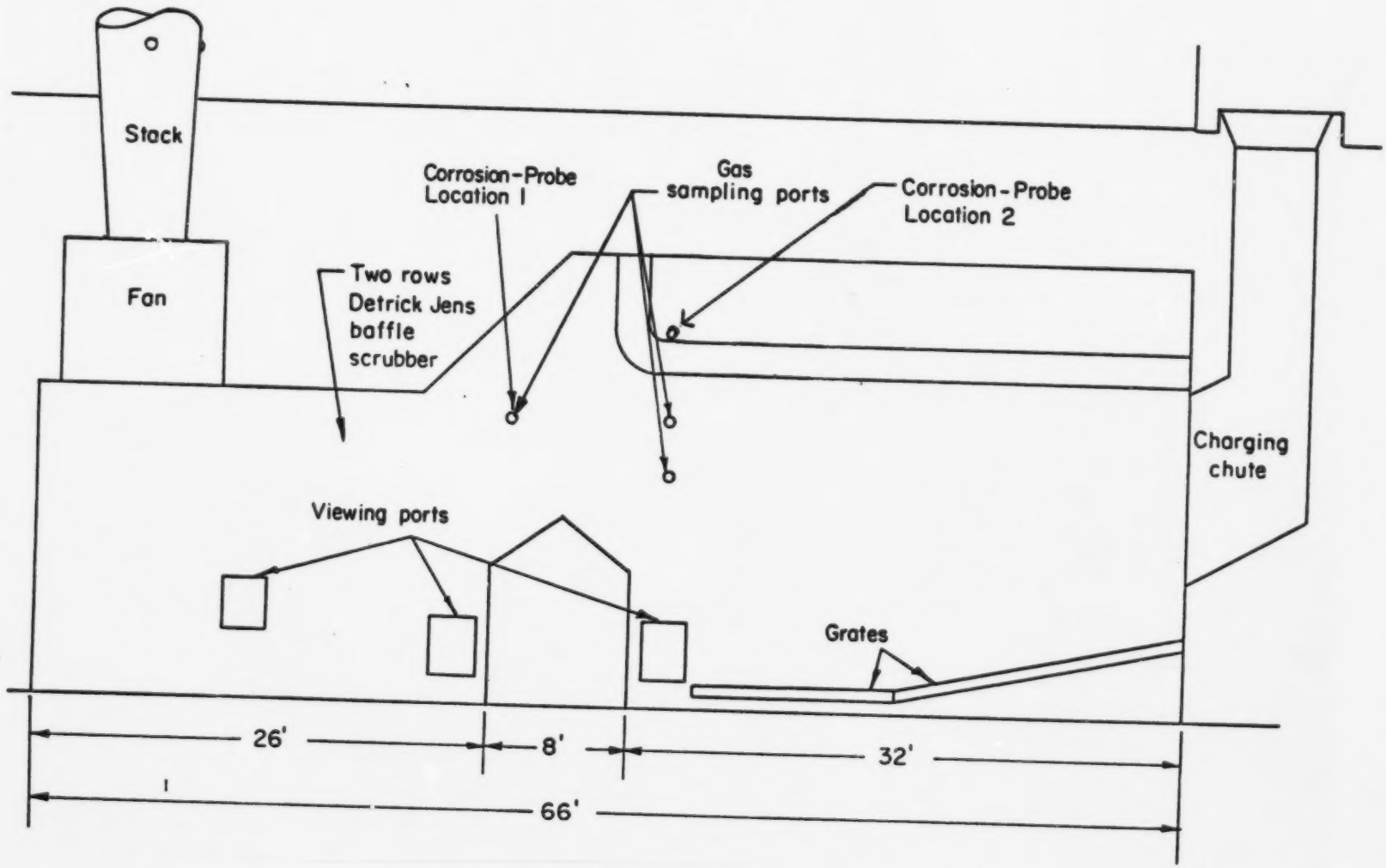

FIGURE 3. SKETCH OF MIAMI INCINERATOR

Scale: 1/8 inch = 1 foot.

Corrosion Rate Analysis

Corrosion rates were determined for each specimen on the five probe experiments by comparing the initial weight with that obtained after removal of the corrosion products. The 34 corrosion specimens were first separated from each other by tapping with a fiber mallet. Then the amount of metal wastage was determined by chemically or electrochemically stripping the residual deposits and scale from each specimen using standard procedures. The specimens of T11 and A106 were stripped cathodically in 10 percent H_2SO_4 containing 1-ethylquinolinium iodide inhibitor, while the stainless steel specimens were descaled in a two-step process: first with 11 percent NaOH and 5 percent $KMnO_4$ at 212 F and then with 20 percent HNO_3 with 2 percent HF at 130 F. These methods of scale removal were employed to avoid attack of the uncorroded substrate metals. Then the weight loss and dimensional changes were measured. Prior to removal of the corrosion products and the deposits, optical and X-ray diffraction studies were made of the scale and deposit on several specimens to determine what phase changes occurred as a result of the type of refuse or the amount of PVC added. Furthermore, the deposits were analyzed for compositional variations in different temperature zones along each probe.

RESULTS

Corrosion Rate Studies

Low Alloy Steels

The effect of temperature on the corrosion rate of the A106 and T11 steels when exposed to hot gases resulting from incineration of refuse to which 0-2 percent PVC was added are shown in Figures 4 and 5, plots of corrosion rate versus temperature for the various amounts of PVC added to the refuse. These data show a significant increase in the corrosion rates of the low-alloy steels with temperature and with the addition of PVC to the refuse. However, the increase in corrosion rate for these steels is not proportional to the amount of PVC added except for specimens allowed to heat to above 1000 F for which the corrosion rate increases linearly with PVC addition. This observation is shown graphically in Figures 6 and 7, plots of isothermal corrosion rate versus the amount of PVC added to the refuse. The addition of even 1/2 percent PVC to the refuse increases the corrosion rate of specimens, held at temperatures between 300 F and 900 F by nearly as much as does the addition of 1 or 2 percent PVC. In this range of specimen temperatures, the addition of 1 percent PVC increased

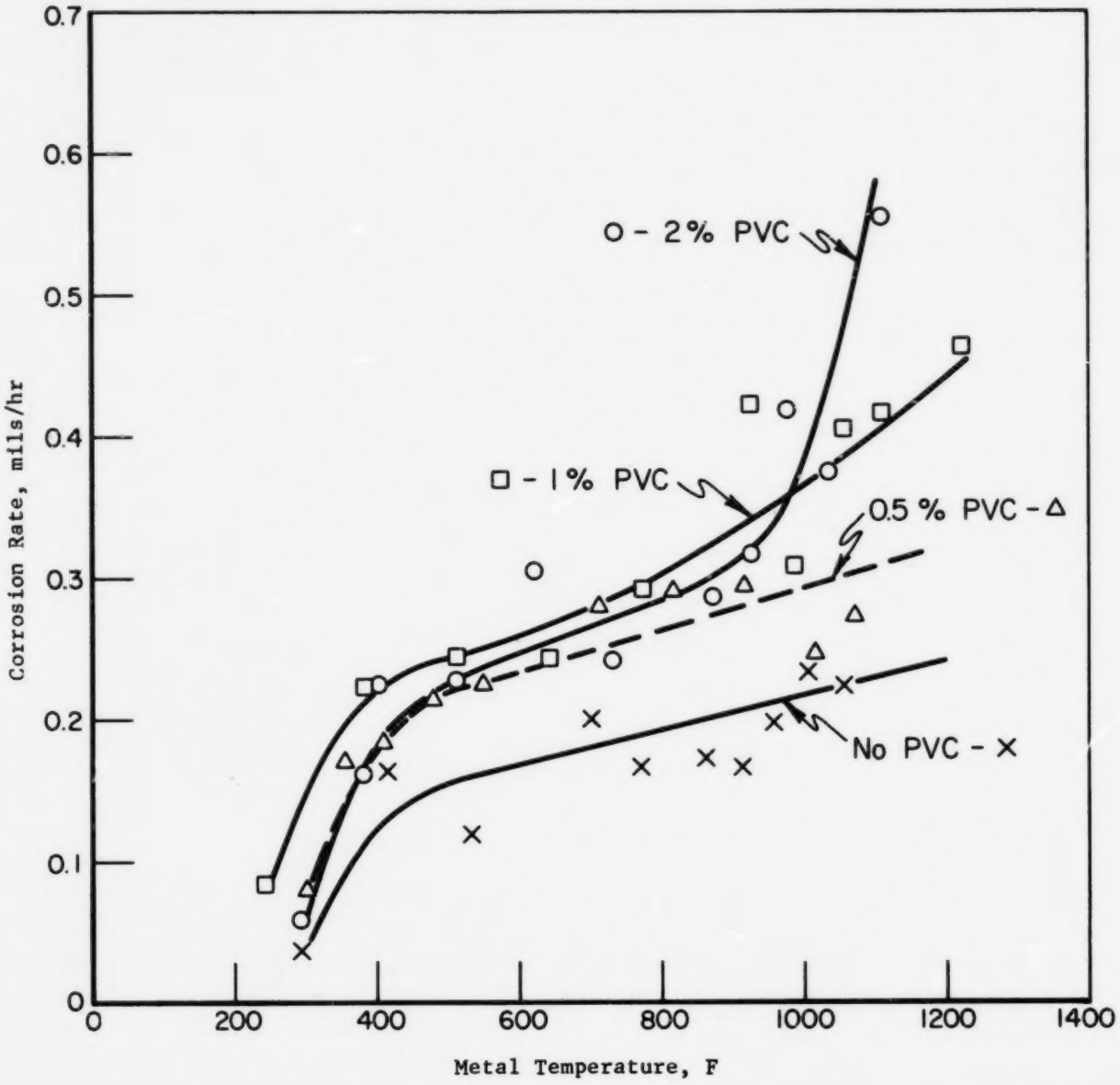

FIGURE 4. CORROSION RATE OF A106 STEEL VERSUS TEMPERATURE FOR DIFFERENT PVC LOADS

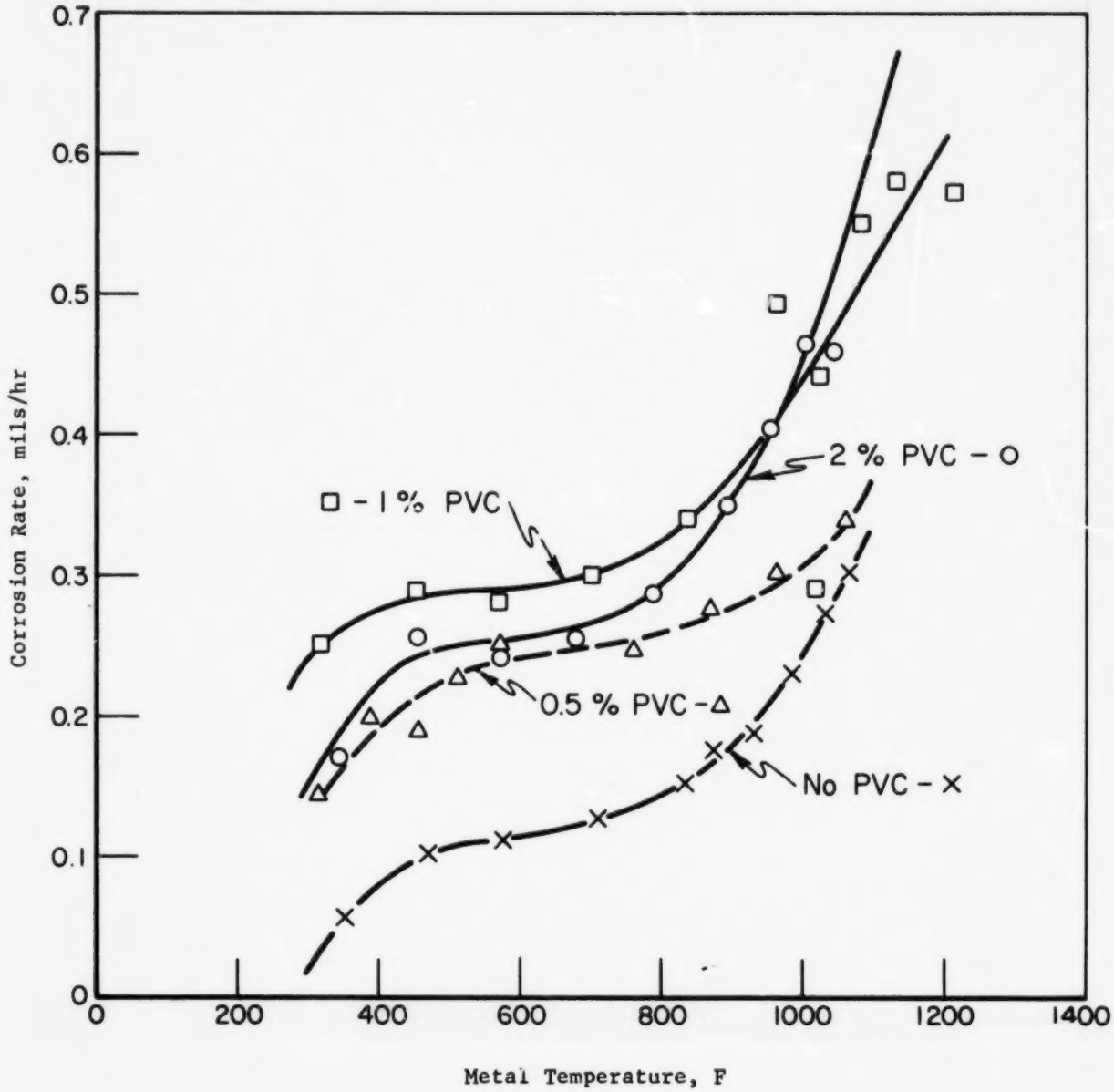

FIGURE 5. CORROSION RATES OF T11 STEEL AS A FUNCTION OF SPECIMEN TEMPERATURE FOR DIFFERENT PVC LOADS

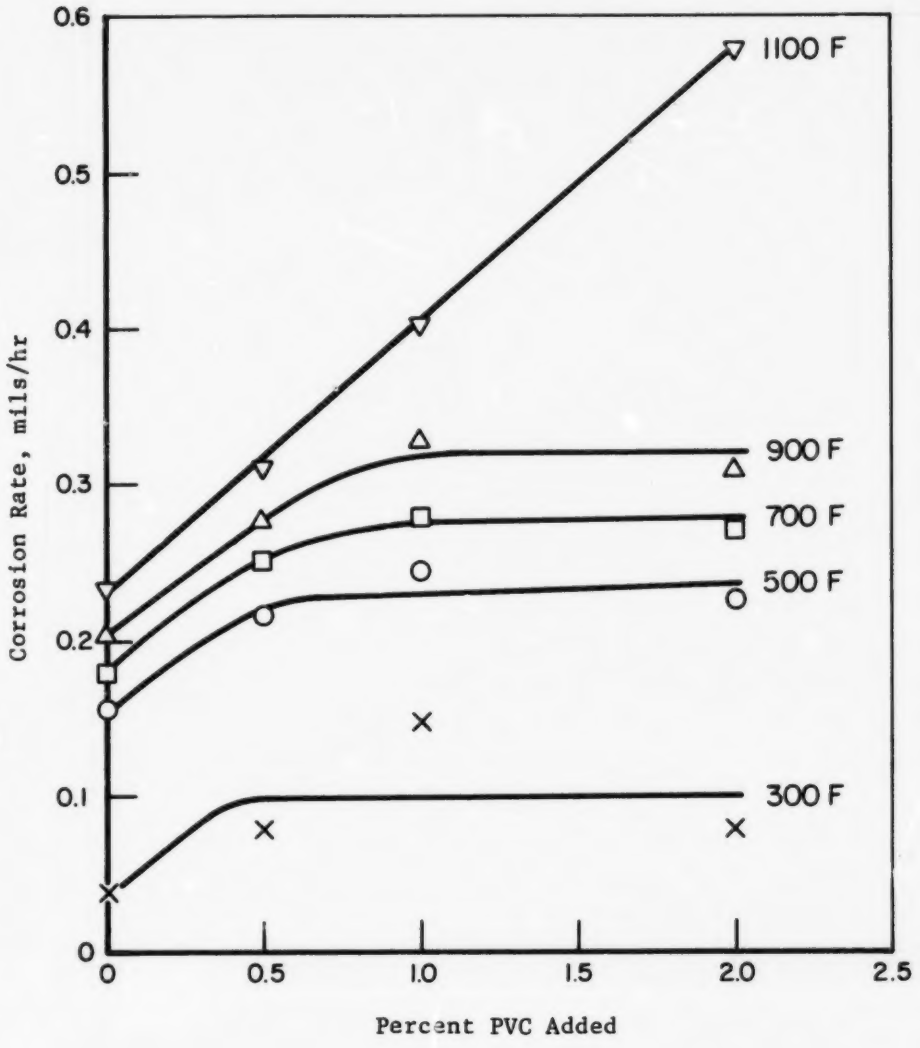

FIGURE 6. CORROSION RATE OF A106 STEEL AT VARIOUS TEMPERATURES VERSUS PERCENT PVC ADDED TO REFUSE

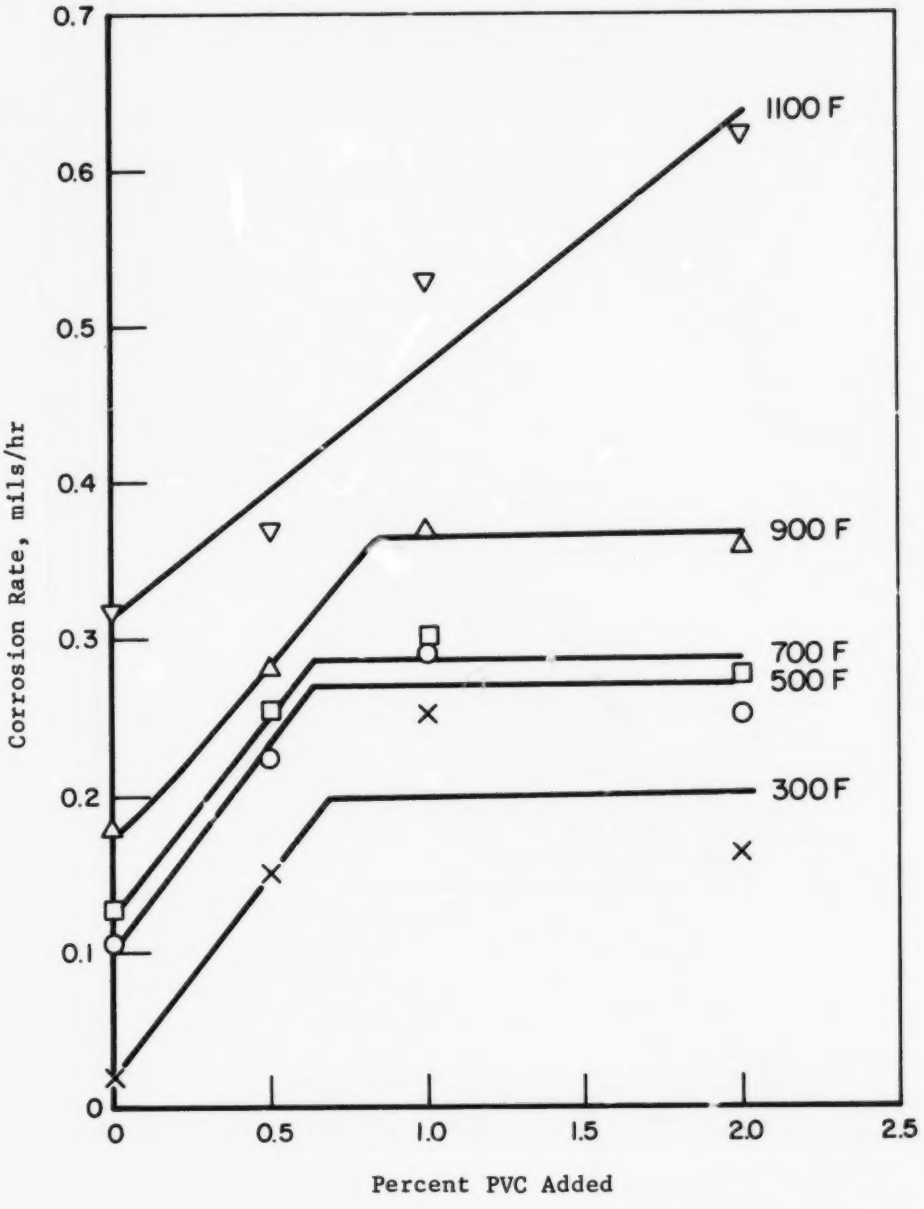

FIGURE 7. CORROSION RATE OF T11 STEEL AT VARIOUS TEMPERATURES VERSUS PERCENT PVC ADDED TO REFUSE

the corrosion rate somewhat more than did the 2 percent addition. However, there were at least two differences in the experimental conditions between Run 17 (2%) and Run 18 (1%):

(1) The incinerator operated 300 F hotter for Run 18.

(2) Probe 17 was exposed to the hot gas of the incinerator for ∽ 20 minutes before an HCl detector registered an increase due to the PVC loading.

An evaluation of these variables was made during the run with Probes 19 and 20, the results of which are shown in Figures 8 and 9. The major effect of incinerator gas temperature, which was 1400 F for Probe 19 and 1550 F for Probe 20, on corrosion rate was found to occur with specimens whose temperature exceeded 800 F. The 150 F hotter gas caused a marked increase in corrosion rate and the difference increased with increasing specimen temperature for both the A106 and the T11 steel. This increase in corrosion rate, because of the hotter incinerator gas, was observed also for the stainless steels as described in the next section. It is evident from Figure 9 that preoxidizing had no significant effect on the corrosion rate of the T11 steel specimens on Probes 19 or 20.

Stainless Steels

All of the high-alloy stainless steels evaluated exhibited much lower corrosion rates than the low-alloy steels even though the stainless steels were, in general, exposed at higher specimen temperatures. There was, as shown in Figures 10 and 11, essentially no effect of specimen temperature or PVC content of the refuse on the corrosion rate for the stainless steels. However, their corrosion rates increased by a factor of 2X-6X when the incinerator gas temperature increased. The effect of gas temperature on the corrosion rates of the stainless steels is shown in the following tabulation:

Alloy No.	Average Corrosion Rate, mils/hr	
	1400 F Gas	> 1500 F Gas
310	0.032	0.170
446	0.050	0.136
825	0.056	0.259
316	0.056	0.203
321	0.109	0.201

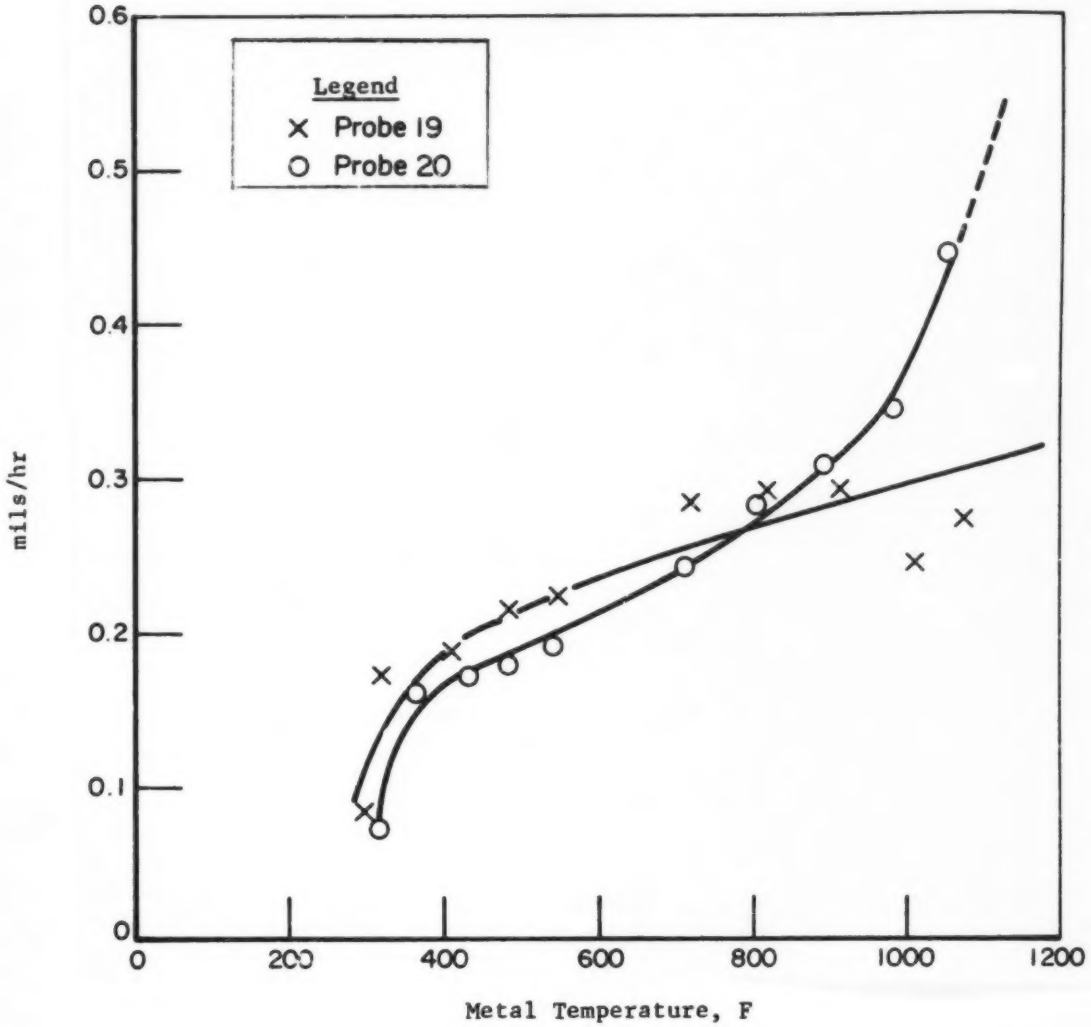

FIGURE 8. EFFECT OF GAS TEMPERATURE ON CORROSION RATE OF A106 STEEL

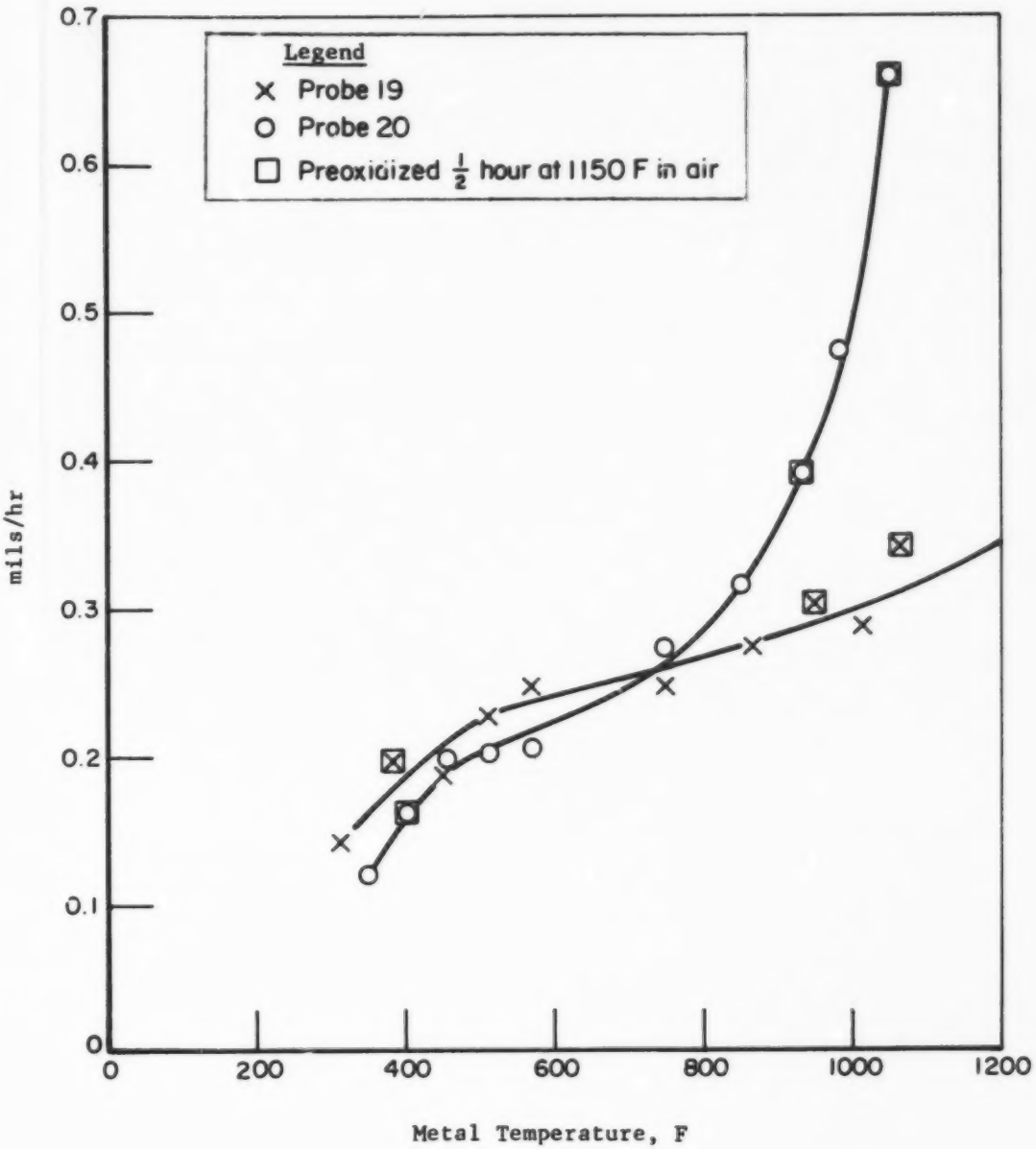

FIGURE 9. EFFECT OF GAS TEMPERATURE ON CORROSION RATE OF T11 STEEL

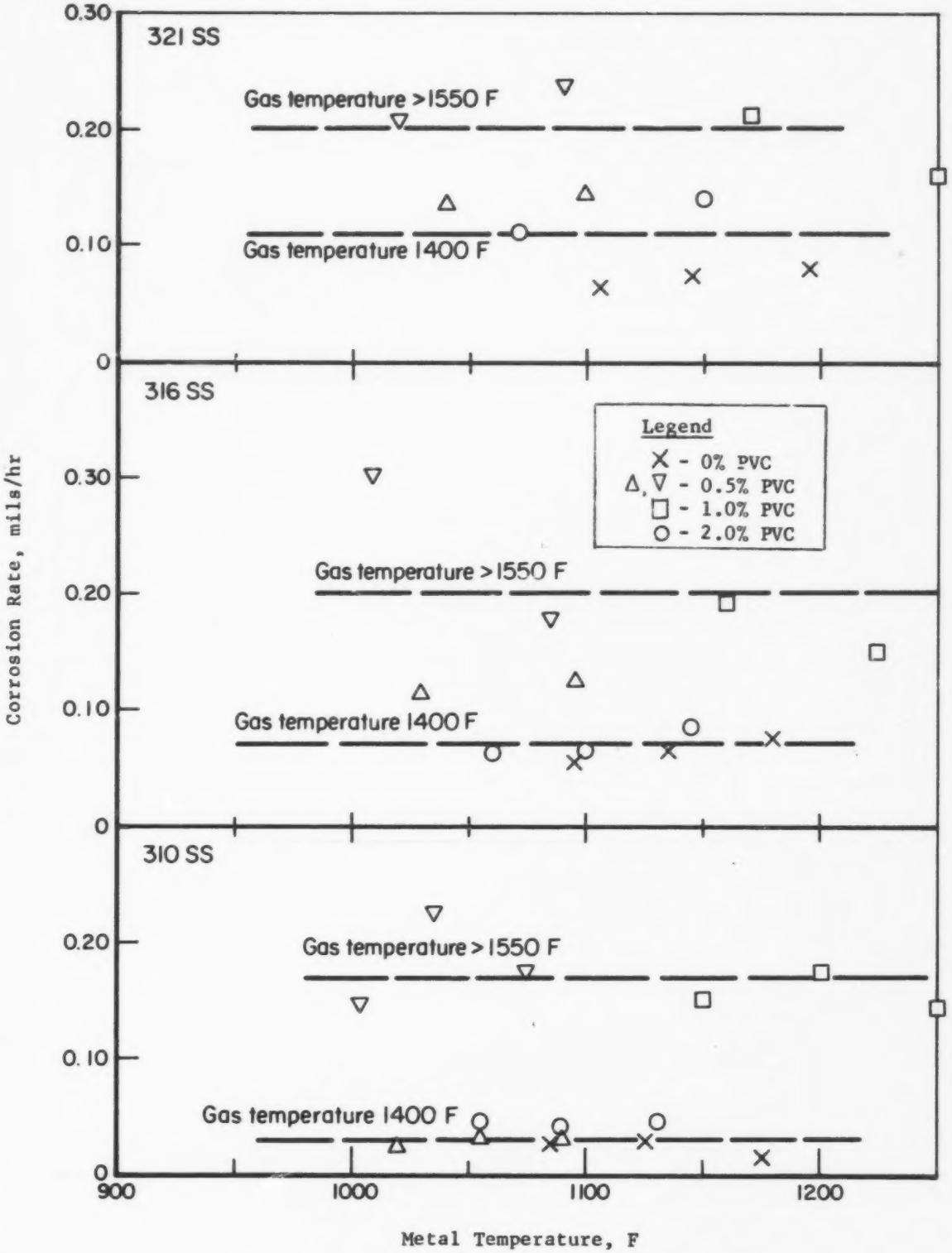

FIGURE 10. CORROSION RATE OF ALLOY INDICATED VERSUS TEMPERATURE

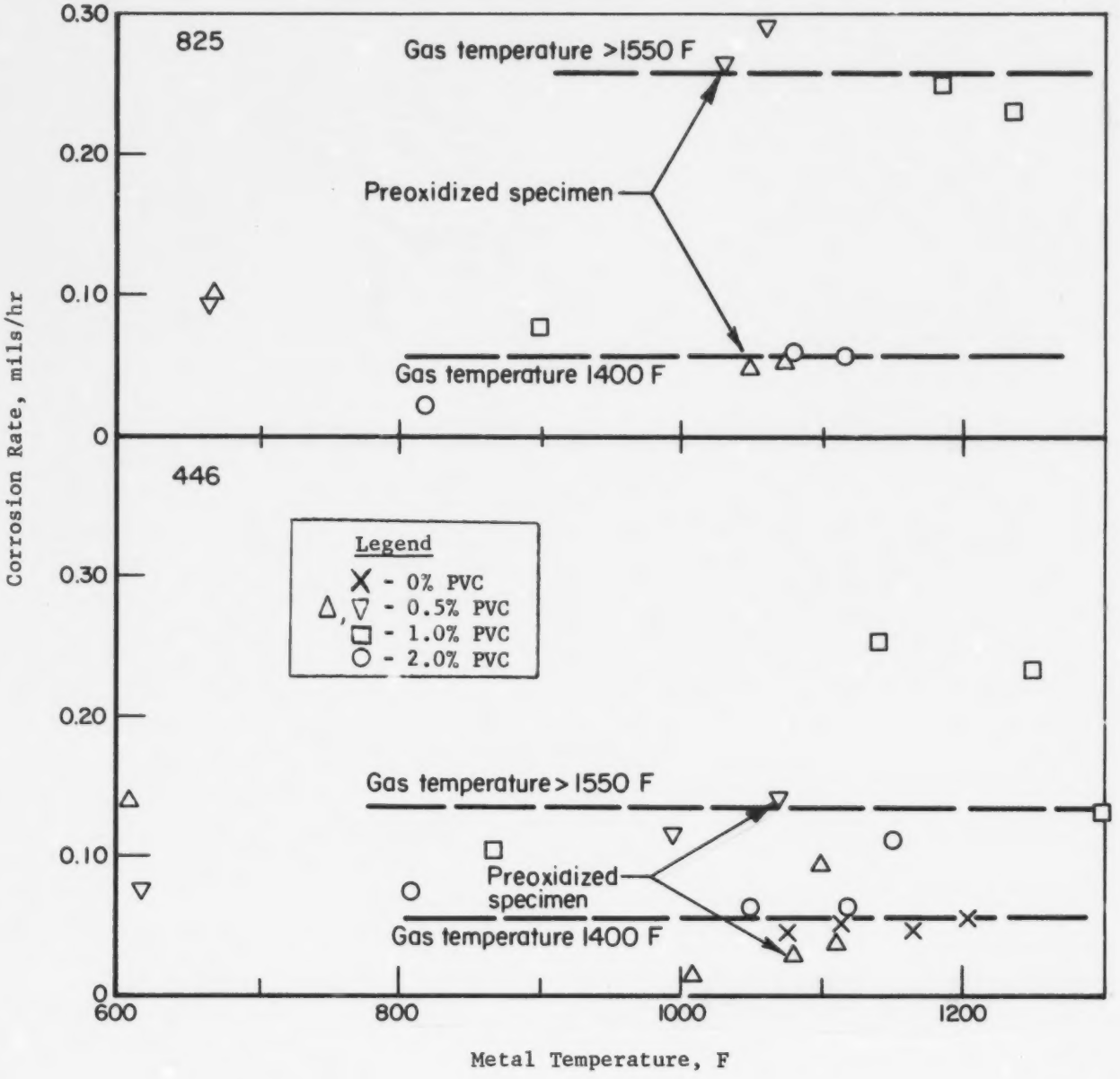

FIGURE 11. CORROSION RATE OF ALLOYS INDICATED VERSUS TEMPERATURE

The major mode of attack is intergranular with some pitting corrosion and grain fallout which produces a grainy (sugar-like) surface. Preoxidation of Alloys 446 and 825 for 1/2 hour in air at 1150 F did not significantly alter the mode or the extent of attack, see Figure 11. Furthermore, no systematic differences in corrosion rates of the high alloy steels were observed that could be related to the amount of PVC added to the refuse. If the heat transfer tubes are located in an incinerator gas whose temperature does not exceed 1400 F, the corrosion rate of the stainless steels would be expected to be 1/6 to 1/10 that of the low-alloy steels depending upon metal temperatures maintained. The high-alloy steels do not suffer as severe a loss in corrosion resistance with increasing metal temperature as is observed with the low alloy steels. However, all steels investigated are very sensitive to incinerator gas temperature when the specimen temperature exceeds 800 F.

Corrosion Product and Deposit Analysis

As the corrosion probes were inserted into the fireside chamber of the incinerator above the effluent end of the grate, the airflow carried sufficient particulate matter to produce a deposit of 1/4 to 1/2-inch thick over approximately 1/3 of the 1-1/4-inch-diameter tube in the 10-hour exposure periods. The remaining 2/3 of the surface contained only a thin deposit and an oxide layer, the oxide layer extending completely around the tube beneath the deposit. The thickness of the oxide layer varied for the various alloys and depended to a lesser extent upon specimen temperature. These layers of deposit and oxide were removed mechanically and collected by temperature zones for optical emission spectroscopy analysis of the metallic element present. The results of optical emission spectroscopy analyses are presented in Table 2. Also included in Table 2 are the results of wet chemical analyses for chlorine and sulfur concentrations and of phase analyses by X-ray diffraction. The chloride concentration in the deposits decreases rapidly with increasing specimen temperature as shown in Figure 12 for the five probes, presumably due to reaction of the chloride with the furnace gases and vaporization. It is interesting to note that the chloride content of the deposit on specimens held at temperatures above 800 F appears to increase linearly with the amount of PVC added to the refuse as shown in Figure 13. However, at the lower specimen temperatures, the chloride in the deposit increases more rapidly between the 0 and 0.5 percent PVC addition than between 0.5 and 2.0 percent addition. Furthermore, at the lower specimen temperature, the amount of chloride in the deposit is lower on probes exposed to the higher temperature (> 1550 F) incinerator gases than on probes exposed to 1400 F gas. Thus, the data points, in parenthesis in Figure 13, fall well below the joined data points.

TABLE 2. RESULTS OF CHEMICAL ASSAY OF DEPOSITS

Elements Detected	Probe 16		Probe 17		Probe 18		Probe 19		Probe 20	
	400 F - 800 F	800 F - 1200 F	400 F - 800 F	800 F - 1200 F	400 F - 800 F	800 F - 1200 F	400 F - 800 F	800 F - 1200 F	400 F - 800 F	800 F - 1200 F
	Concentration, weight percent									
Na	7.5	7.5	5.0	4.0	6.5	4.5	6.9	7.5	6.9	7.5
K	11.5	19.0	8.5	8.0	10.8	7.5	3.7	3.6	4.4	3.7
Ca	6.0	7.5	7.5	10.0	10.8	12.5	6.4	12.5	6.7	12.5
Mg	2.0	3.0	2.5	3.5	3.3	3.0	1.0	2.0	0.6	2.0
Pb	2.5	1.0	2.0	0.5	3.0	0.3	3.2	0.3	3.1	0.3
Zn	6.0	6.5	6.0	4.5	6.5	4.5	6.9	7.5	6.9	7.5
Fe	5.0	6.0	6.5	6.0	8.5	6.3	12.3	7.1	12.6	6.6
Al	14.5	12.5	7.5	8.5	6.5	7.3	3.0	5.6	3.3	6.0
Si^{-}	4.5	5.5	6.0	6.5	7.5	7.5	3.4	7.5	3.9	7.5
Cl^{-}	4.0	1.5	11.5	4.5	5.8	1.3	7.6	3.6	4.8	1.9
$S^{=}$	8.0	9.0	6.5	5.5	6.9	8.6	7.8	8.3	4.9	7.6
O_2	Balance		----		----		----		----	
	Phase Studies on Deposits									
	$CaSO_4$ + SiO_2 + $K_3Na(S)_4)_2$		$CaSO_4$ + $NaCl$ + KCl + SiO_2		$CaSO_4$ + $NaCl$ + SiO_2		$CaSO_4$ + Ca_2SiO_4 + $NaCl$		$CaSO_4$	$CaSO_4$ + SiO_2 + Ca_2SiO_4
								$CaSO_4$ + Ca_2SiO_4 + $K_3Na(SO_4)_2$		

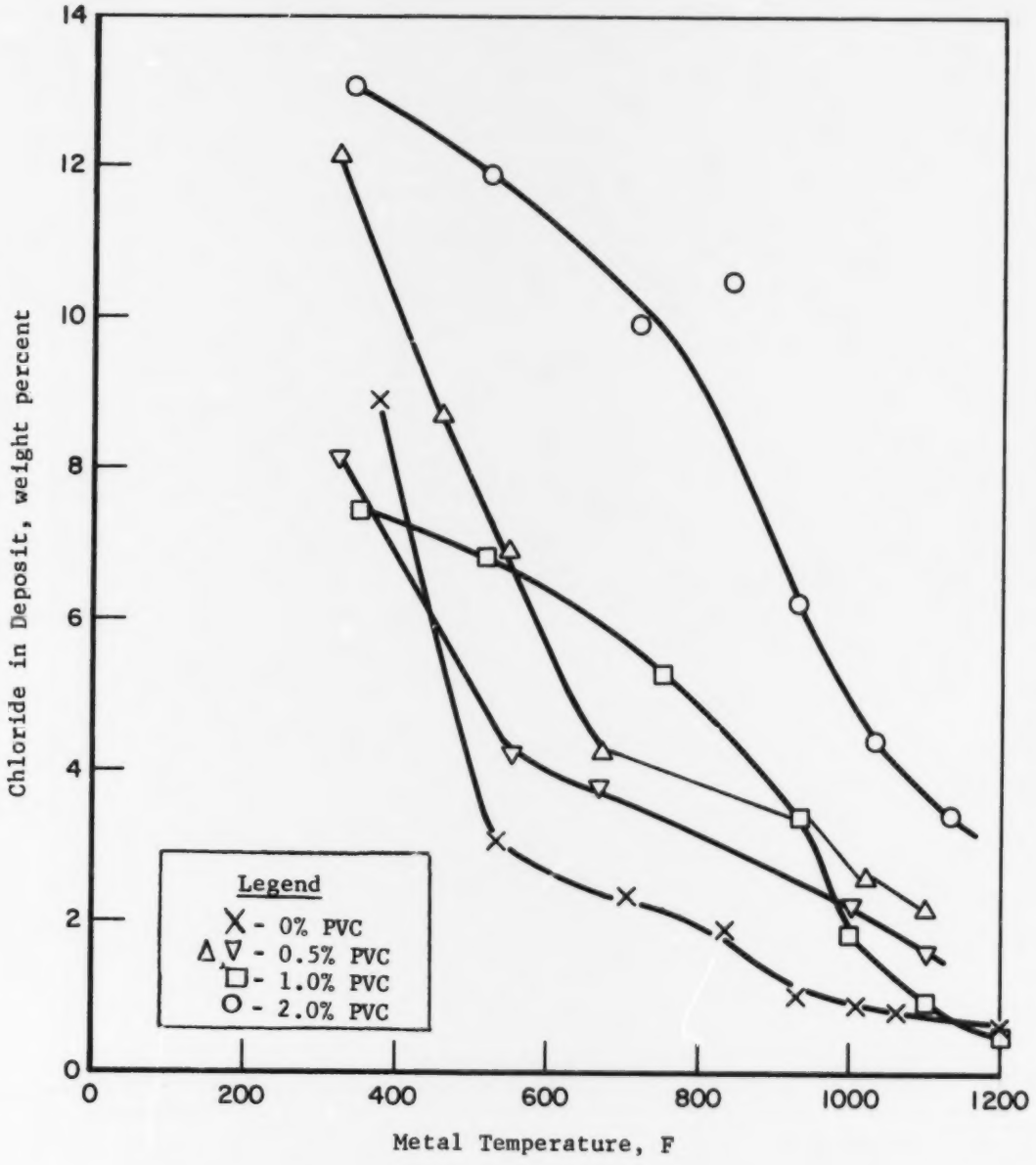

FIGURE 12. CHLORIDE CONCENTRATION IN DEPOSIT ON PROBES VERSUS METAL TEMPERATURE

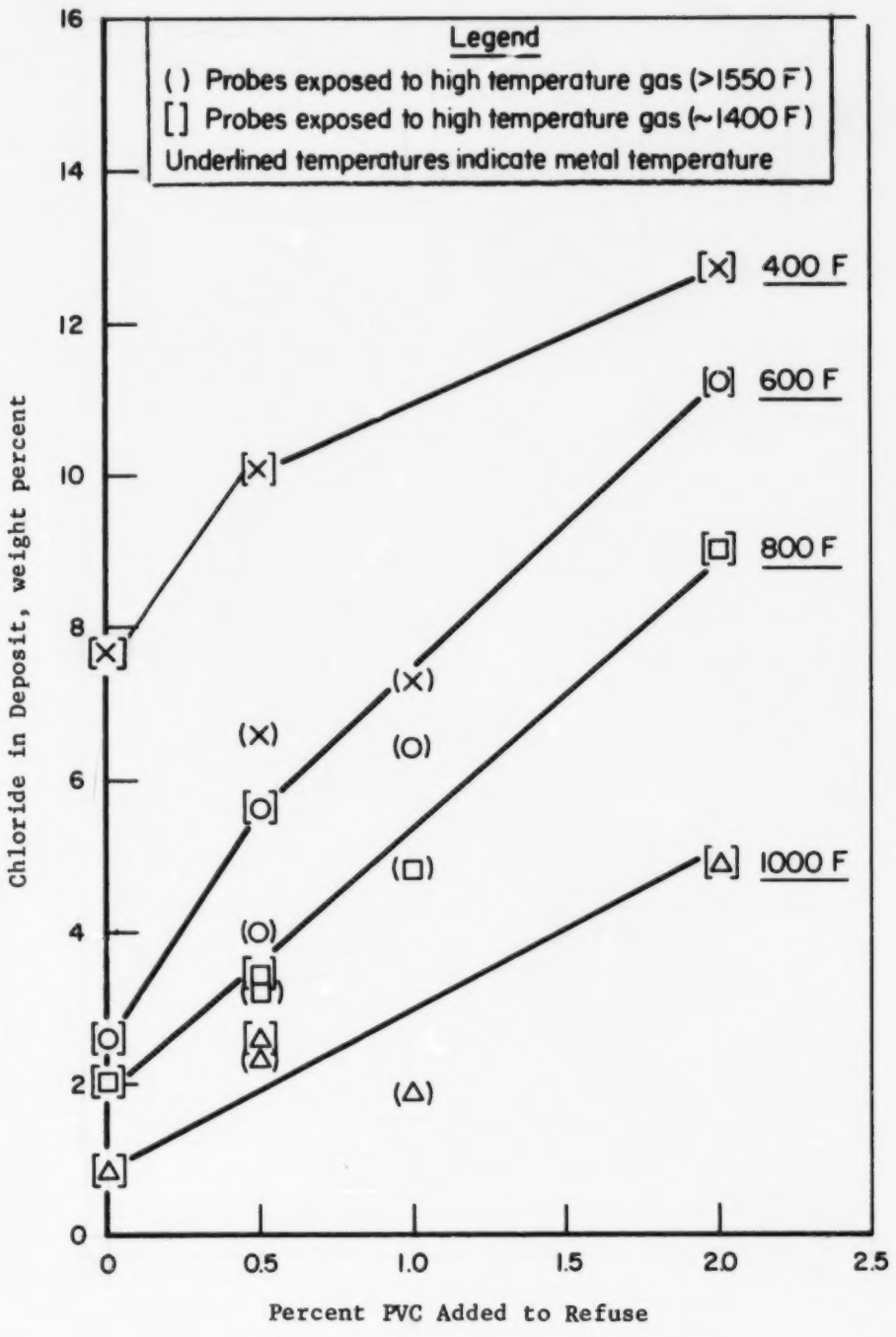

FIGURE 13. CHLORIDE CONCENTRATION IN DEPOSIT VERSUS AMOUNT OF PVC ADDED TO REFUSE

The sulfur content of the deposit is seen, in Table 2, to be more uniform and perhaps slightly higher at the higher metal temperatures. Several specimens representing various temperature zones were reserved for X-ray diffraction phase analysis of the deposits and of the scale layers between the oxide and the substrate metal. Phase studies of the deposits are presented in Table 2 while the phase studies on the scale beneath the oxide layer are presented in Table 3. The oxide layer contains a mixture of Fe_3O_4 and Fe_2O_3 which forms a semipermeable barrier layer between the deposit and the scale. The results presented in Table 3 on phases formed between the substrate metal and the oxide layer show the mechanism of attack to be similar to those presented in our summary report under the previous (EP000325) grant. The major difference in the low-temperature zone (400-800 F) is that the ferrous chloride forms a continuous layer when PVC is added to the refuse while normal refuse produces a discontinuous layer on the metal substrate. This increase in the amount of ferrous chloride formed when PVC is added to the refuse also contributes to the corrosion in the high-temperature (800-1200 F) zone. Here the chloride melts, agglomerates, and probably vaporizes exposing clean metal for the sulfide reaction. During these changes in the chloride phase, the semiprotective oxide layer on low alloy steel is undoubtedly partially destroyed. However, the alloy oxide that forms on the stainless steels is more resistant to this chloride attack.

Incinerator Gas Composition

Previous sections of this report have discussed the effect of refuse composition on the corrosion of metal and the role of deposits and scale upon the corrosion mechanisms. The composition of the deposits is partially controlled by the noncombustible portion of the refuse, by the gaseous species in the incinerator, and by the temperature of the substrate and of the surrounding incinerator temperature. At probe Location 1 of Figure 3 and to some extent at Location 2, the off gases of the refuse are diluted with the overfire air, the amount of which varies to maintain a temperature of 600 F at the ID fan. As a result of variations in the amount of overfire air the composition of the gas will fluctuate.

The composition of the refuse varies resulting in some changes in off gas composition when refuse samples are combusted in the laboratory. As shown in Table A-2 of the Appendix, the percent chlorine in the off gas from grab samples of refuse differs by a factor of over 2X (0.34% to 0.74%). Table 4 shows fluctuations in gas composition for Probe Runs 16, 17, and 18 where gas samples were collected in two batches during each exposure period. Because of these wide fluctuations, the gas was sampled throughout Run 19/20. As would be expected, however, the amount of HCl in the incinerator gas (0.03 to 0.09%) is found to be 1/7 to 1/12 that evolved from the refuse due to dilution by over- and under-fire air.

TABLE 3. PHASE STUDIES OF SCALE NEXT TO METAL

Probe Number	Phases By Layers Between the Metal and the Oxide Layer	
	400 F - 800 F	800 F - 1200 F
16	$FeCl_2 \cdot 4H_2O$ - FeOOH - FeS (≠)	($FeCl_2 \cdot 4H_2O$ + FeOOH + FeS) (*)
17	$FeCl_2 \cdot 4H_2O$ - KCl	($FeCl_2 \cdot 4H_2O$ + FeOOH + NaCl) (*)
18	As 17 - no evidence of sulfide layer (†)	As 17 (†)
19	$FeCl_2 \cdot 2H_2O$ - $FeCl_2 \cdot 4H_2O$ - FeOOH - FeS (≠)	($FeCl_2 \cdot 2H_2O$ + FeOOH + FeS) (*)
20	As 19	($FeCl_2 \cdot 2H_2O$ + FeS) (*)

(*) Phases in this temperature zone do not form successive layers but are present in discrete areas.

(†) Analyzed by optical method.

(≠) This FeS is found at the interface between the ferrous chloride and the oxide that separates the deposit from the scale.

TABLE 4. INCINERATOR GAS ANALYSES TAKEN DURING CORROSION RATE STUDIES

Component	Probe Run Number								Prior Analyses(‡)
	16(*)		17(*)		18(*)		19-20(†)		
	1	2	1	2	1	2	1	2	
HCl, ppm measured	289	458	545	1180	548	1260	583		5-300
HCl, ppm corr. 12% CO_2	1650	4580	1090	4720	1644	3780	2000		
SO_2, ppm measured	185	29	56	82	88	471	57		0-300
SO_2, ppm corr. 12% CO_2	1850	580	168	328	264	2260	195		
SO_3, ppm measured	2	25	4	14	21	6	8		
SO_3, ppm corr. 12% CO_2	20	500	12	56	63	29	27		
NO_x, ppm measured	35	7	73	38	70	55	32	80	4-138
NO_x, ppm corr. 12% CO_2	200	140	219	152	210	264	96	320	
CO_2, percent	2.1	1.2	6.0	4.0	4.0	2.5	4.0	3.0	4-12
O_2, percent	18.7	17.8	15.2	17.2	17.5	11.5	17.2	17.2	
H_2O, percent	12.3	4.8	6.9	8.0	10.0	11.7	5.3		

(*) Gas samples at two times 1 hour each for these runs.

(†) Gas samples throughout the period of this run.

(‡) Values obtained previously at Miami County, Oceanside. and Norfolk.

Sampling and Analysis Methods

HCl samples were collected by two methods: (1) a standard EPA sampling train consisting of a probe, heated filter, and two impingers each containing 100 ml of 5 weight percent NaOH; the chloride analysis was done by titration with silver nitrate after acidifying the solution; and (2) a continuous chloride monitoring system based on a chloride electrode (Orion). The sampling system consisted of a probe, heated filter, and a bubbler containing 500 ml of acetate buffer solution (pH 5.5) in which the chloride electrode was immersed. The electrode output was recorded on a strip chart to provide continuous readout of the potential change from a chloride sensitive half-cell in the solution. Changes in HCl concentration in the furnace gases could be detected readily by this method and the actual concentration of HCl was determined by comparison with standard chloride solutions. Some discrepancies were noted between chloride concentrations found by the two methods. These differences are believed to result from relative absorption efficiencies in the two systems.

Sulfur oxides were collected using a standard EPA train consisting of a probe, heated filter and three impingers. The first impinger contained 100 ml of 80 percent isopropanal to absorb SO_3. The other two impingers each contained 100 ml of 3 percent H_2O_2 to absorb SO_2. Both the SO_2 and the SO_3 were then determined by titration of the respective absorption solutions with barium perchlorate, using thorin as the indicator.

Nitrogen oxides were collected by taking a grab sample in a 3-liter evacuated flask which contained 25 ml of 3 percent H_2O_2 acidified with H_2SO_4. The analyses were made by the standard phenol disulfonic acid method.

Combustion-gases (carbon dioxide, oxygen, and carbon monoxide) were determined by standard Orsat methods and Fyrite analyzers. The water content was measured by condensing the moisture and determining its volume.

Results of Gas Analysis

The data presented in Table 4 illustrate large hourly as well as daily variations in nearly all of the components of the incinerator gas. Although the average HCl contents from the two samplings of the gas for Runs 17 and 18 and the amount obtained in the all-day collection of Run 19/20 are higher than for Run 16, they do not appear to be in proportion to the amount of PVC added to the refuse. On the other hand, the chloride content in the deposits on the corrosion probes do vary with the amount of the PVC addition to the refuse (see Figures 12 and 13). Thus, it would appear that a

large fraction of the HCl evolved from the PVC combines with the metallic elements or their oxides to form inorganic chlorides rather than remaining as HCl gas. Thus, the HCl content of the incinerator gas is believed to be affected by other compositional variables in the refuse.

A summary of the results of the two methods of collecting gas for HCl analyses are presented in Table 5. It is evident here that Method 1, the dual impinger alkaline solution, was a more efficient collector than Method 2, which consists of a single bubbler with an acid solution. However, the latter gave an immediate response and could be used to determine when the gases from the combustion of PVC had reached the corrosion probe area of the incinerator. The result of Method 1 (average 860 ppm) for the 2 percent PVC addition (Run 17) compares very well with the 816 ppm obtained by Kaiser[3] when 2 percent PVC was added to the Babylon Incinerator. The higher HCl content in Run 18 when only 1 percent PVC was added is undoubtedly caused by other compositional variables in the refuse. The other constituents of the gas environment, obtained on this program, are consistent with previous analyses of furnace gas composition at Miami County, Oceanside, and Norfolk incinerators.

DISCUSSION OF RESULTS

The results of corrosion rate, scale, and deposit studies illustrate that the PVC content of refuse will have a marked effect on the life of materials for heat recovery systems in municipal incinerators. The corrosion of low alloy steels A106 and T11 may be divided into three temperature zones: < 400 F, 400-800 F, and 800-1200 F. In the low-temperature zone, the corrosion rate decreases rapidly with decreasing metal temperature while from 400-800 F, the corrosion rate is essentially constant. Between 800 F and 1200 F, the corrosion rate increases rapidly with metal temperature. The behavior in the 800-1200 F zone is very similar to that observed[6] with high (0.75%) chloride coal having relatively low (0.9%) sulfur, but the corrosion rate of 0.07 mil/hr in 100 hour exposures at 1200 F when coal was burned is approximately 1/5 that observed in an incinerator with normal refuse and 1/10 that with a 2 percent PVC addition. This difference, however, may be attributed in part to the furnace gas temperature at the location of the specimens. As shown in the present study, an increase of 150 F (1400 F to 1550 F) in gas temperature can increase the corrosion rate of specimens maintained at 1100 F, by internal cooling, by a factor of as much as 6X. If, however, the specimens are held to below 800 F, the higher furnace gas temperature does not affect the corrosion rate.

Although there is a significant (1.5-2X) increase in corrosion rate at all temperatures by the addition of 0.5 percent PVC to the normal refuse, the addition of larger (1% and 2%) amounts of PVC does not continue to increase the corrosion rates appreciably, when the

TABLE 5. SUMMARY OF RESULTS ON HCl CONTENT OF GAS

Method (*)	HCl Content, ppm Probe Run Numbers			
	16	17	18	19/20
1	370	860	904	583
2	210	801	448	71
3		343	375	98
4		364	392	104

(*) Methods 1 and 2 are described previously while Method 3 involved titration of the solution from 2 and Method 4 was X-Ray fluorescence analysis of the solution from 2.

metal temperatures are held in the range of 400 to 800 F. However, the corrosion rates of A106 and T11 steels increase linearly with PVC content for higher (1100 F) metal temperatures. Alexander[7] observed the air oxidation of Type A213-T22 steel at 1100 F to increase a factor of $\sim$ 2X by coating the steel with a 10 percent $NaCl-Na_2SO_4$ mixture while in the present study, increasing the PVC content of the refuse by 2 percent increased the chloride content of the deposit to 4 percent and the corrosion rate of T11 at 1100 F by a factor of 2.5X from 0.22 to 0.55 mil/hr. While the oxidation rates observed by Alexander are 1/100 those obtained in the present work, it is important to note that her specimens were exposed to air rather than to incinerator gases.

It appears that the corrosion mechanism is different above 800 F from that below 800 F. This is confirmed by the composition of the scale layer next to the metal in that below 800 F the layer is composed of ferrous chloride while above 800 F, there is a mixture of ferrous sulfide with pools of ferrous chloride on the metal substrate. The initial increase in corrosion rate of the low-alloy steels with a small increase in PVC content of the refuse for temperatures below 800 F is attributed to the development of a continuous layer of ferrous chloride on the metal surface. At higher temperatures, however, the role of PVC in the refuse is one of maintaining sufficient chloride ion to destroy the oxide films and thus allow sulfide corrosion to occur, even though the sulfur content of refuse is low compared to that of coal. It is evident from analyses of the deposit that the amount of chloride retained on the corrosion probes decreases rapidly with increasing metal temperature. Perhaps volatization of ferrous chloride is also contributing to the high corrosion rates in the upper temperature zone of the corrosion probe. This would account for the drastic corrosion observed by Heimburg[1] during the burning of PVC.

Unlike the low-alloy steels, the corrosion rates of the stainless steel were essentially unaffected by specimen temperature (800-1200 F) or by the PVC content of the refuse. However, the corrosion rates for the stainless steels were increased considerably (2X-6X) by an increase of 150 F in gas temperature. This increase in corrosion rate with gas temperature was observed for low-alloy-steel specimen also when the metal temperature exceeded 800 F, but was not observed at lower (400-800 F) metal temperatures. The lack of an effect of PVC content in the refuse on the corrosion rates of the stainless steels is undoubtedly due to the type of oxide formed and the low retention of chloride in the deposits in the high temperature zone on the probe. Alternatively, the base chloride content of refuse may be sufficient to reach an equilibrium in corrosion rate versus PVC content as was observed in the low temperature zone for the low-alloy steel. Alexander[7] found the oxidation rate of 316 stainless steel coated with a 10 percent $NaCl-NaSO_4$ mixture to be nearly the same as that with 20 percent $NaCl-Na_2SO_4$ mixture while with lower amounts of NaCl the oxidation rates increased linearly with NaCl content. The results presented in this study would indicate that the

corrosion rates of the stainless steels, in the incinerator gases, do not increase with chloride content of the deposit above about 1-2 percent chloride which is obtained with normal refuse. The corrosion rate of 0.045 mil/hr for 316 stainless steel at 800-1200 F in 1400 F incinerator gas is about 30X that obtained by Alexnader for the oxidation rate of 316 stainless steel coated with a 2 percent $NaCl-Na_2SO_4$ mixture. This large difference is undoubtedly attributable to differences in gas composition between air and the incinerator gas as previous laboratory studies at Battelle[8] have shown that the combination of mixed gases and mixed (1% $NaCl-Na_2SO_4$) salts do produce an attack of stainless steels at 1000 F nearly equivalent to that observed in the present incinerator study. In the above mentioned Battelle investigation, it was shown that both the mixed gas and mixed salt environments were essential to approach the magnitude of corrosion produced in an incinerator. Perhaps if gas temperatures had been increased to that (1400 F) of the incinerator while maintaining specimen temperature, the corrosion rates would have been the same. In the case of the stainless steels, however, the major mode of attack is intergranular which has been shown[9] to be accelerated by the presence of chloride ions, particularly at temperatures above 1000 F.

CONCLUSIONS

(1) The corrosion rates of the carbon and low-alloy steels increase sharply for small (0.5%) additions of PVC to municipal refuse.

(2) Larger (1% and 2%) additions of PVC does not affect the corrosion rate of low-alloy steels at temperatures below 900 F but increases the corrosion rate in direct proportion to the amount added for metal temperature above 900 F.

(3) The general corrosion rates for the stainless steels are not affected by either PVC content of the refuse or metal temperature up to 1200 F.

(4) The corrosion resistance of the stainless alloys to the fireside environment of municipal incinerators decreases in the order: 310, 446, 825, 316, and 321.

(5) All of the stainless alloys suffer grain boundary attack and are susceptible to stress corrosion cracking with the possible exception of 446.

(6) Both the low-and high-alloy steels suffer accelerated (2X to 6X) corrosion rates in the 900-1200 F metal temperature range when the gas temperature is increased from 1400 to 1550 F.

(7) The mechanism of attack at metal temperatures below 800 F is essentially a chloride reaction with the substrate metal while at metal temperatures above 800 F, the attack is predominantly a chloride assisted sulfide reaction.

(8) The chloride content of the deposit increased with PVC content of the refuse, but decreases with increasing specimen temperature and with increasing furnace gas temperature.

(9) The sulfur content of deposits is greater on specimens from the high-temperature zone. Furnace gas temperature has no appreciable affect on the sulfur content of the deposit.

(10) The chlorine (HCl) content of the furnace gas is increased by PVC addition to the refuse, but is not proportional to the amount added.

(11) Based upon the above conclusions, it is further concluded that none of the alloys investigated would provide satisfactory service in fireside heat recovery systems for municipal incinerators.

ACKNOWLEDGEMENTS

The assistance of Mr. R. C. Thurnau and Mr. R. Loebker, of the Solid Waste Research Division, Environmental Protection Agency, in the analysis of types and composition of refuse and their encouragement in the conduct of these corrosion studies were very valuable throughout the investigation. In addition, the cooperation of Mr. R. Karnehm, Superintendent of the Miami County Incinerator at Troy, Ohio, in providing a facility and record of refuse received was a considerable asset to the program. Battelle personnel contributing special assistance to the program were Mr. E. White, Mr. W. Stiegelmeyer, and Mr. J. Faught.

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APPENDIX

ANALYSES OF TYPES AND COMPOSITION OF REFUSE

The hetrogeneous nature of the refuse burned at the Miami County incinerator results from the sources which includes a number of industries plus municipal refuse from surrounding communities. The ratio of municipal to industrial refuse received for the week of June 26, 1972, was 2.08 with a total 878 tons (593 tons municipal and 285 tons industrial). As the amount of each received differs daily, the M/I ratio varied from 0.65 to 4.42. Wednesday, June 28, 1972, had nearly the maximum in the amount (187 tons) received and the M/I ratio was 2.03. Based upon this inspection of the records at the Miami County incinerator, Wednesday was selected as the day of the week to conduct corrosion experiments to evaluate the effect of PVC on the corrosion of water wall materials.

The program consisted of four runs with five corrosion probes and exposure times of 10 hours each. The refuse received on the days of these runs is tabulated below:

Run		Weight of Refuse, tons			
No.	Day	Municipal	Industrial	Total	Ratio
16	8/9/72	117.5	53.5	171.0	2.10
17	9/13/72	82.5	57.0	189.5	1.45
18	10/25/72	86.5	62.0	148.5	1.40
19/20	12/13/72	110.0	40.5	150.5	2.72

As the incinerator operates on a 24-hour-a-day basis, approximately 10/24 of the refuse received was burned during the 10-hour exposure period. No PVC was added during Run 16 to provide baseline corrosion rate data for these short time experiments. For Runs 17, 18, and 19/20, PVC was added to each grapple load fed into the incinerator. Approximately 160 grapple loads were fed into the incinerator in the 10-hour exposure periods. As a grapple load weighs $\sim$ 1000 lbs the amounts of PVC added were 20 lbs (2%), 10 lbs (1%), and 5 lbs (1/2%) per grapple load for the Runs 17, 18, and 19/20, respectively.

In addition to the above catorgization of refuse, Mr. R. Thurnau and Mr. R. Loebker with the assistance of various Battelle staff made a hand sort of a grab samples taken on each of the above dates. These grab samples were taken at approximately 10:00 AM from the refuse pit by the crane operator and placed on the floor adjacent to the pit. The refuse was sorted into 12 categories, weighed and percentages calculated as shown in Table A-1. The total weight of the grab samples ranged from 303 to 785 lbs (0.2-0.5% of the refuse burned in 10 hours). A small but proportional amount of each category was taken to the Solid Waste Research Laboratory in Cincinnati for analysis. The categories were remixed and ground to 4.0-and 0.5-mm size particles for analysis of the following properties:

(1) Moisture content.

(2) Ash content.

    (a) Composition of ash.

(3) Volatiles content.

    (a) Composition of volatiles.

(4) Heat content.

The results of these analyses, as presented in Table A-2, were supplied by Mr. Loebker except for the element analyses on the ash which were made at Battelle by optical emission spectroscopy (OES). The amounts of the various elements found in the ash are very similar in alkali metal content to that found in the deposit, see Table 2 of this report. However, the deposits had significantly higher concentrations of iron, zinc, and lead and considerably more chloride and sulfate than the ash. This suggests that the cold wall of the corrosion probe tends to preferentially retain some materials. The higher iron content in the deposit is probably due to oxidation of the probe.

TABLE A-1. TYPE OF REFUSE IN GRAB SAMPLE (Percent)

Category	Run 16 Aug. 9, 1972 Wet	Run 16 Aug. 9, 1972 % H_2O(†)	Run 17 Sept. 13, 1972 Wet	Run 17 Sept. 13, 1972 % H_2O(†)	Run 18 Oct. 25, 1972 Wet	Run 18 Oct. 25, 1972 % H_2O(†)	Run 19-20 Dec. 13, 1972 Wet	Run 19-20 Dec. 13, 1972 % H_2O(†)	Other Investigation(*)
Cloth	2.07	32.81	2.53	25.48	1.75	10.54	2.1	21.10	2.9
Plastic	3.06	28.35	5.57	14.08	4.59	16.14	2.6	17.44	2.4
Food	17.78	54.56	3.08	58.91	15.25	66.44	9.1	59.13	10.6
Yard and Garden	10.00	62.53	0.04	----	18.10	58.37	1.4	39.05	5.7
Wood	5.29	7.60	2.76	11.92	1.06	12.27	5.4	19.13	3.4
Paper	20.58	36.65	62.05	30.90	24.48	34.84	35.5	26.95	26.2
Corrugated Paper Boxes	13.82	14.61	5.79	29.62	4.27	26.00	2.0	16.08	13.1
Newspaper	2.55	15.18	2.30	39.35	9.4	41.15	11.9	16.04	11.4
Metal	12.87	9.42	6.19	1.95	7.49	8.43	6.4	3.47	8.5
Glass	8.60	0.22	4.16	1.37	5.97	1.95	8.4	0.37	9.4
Magazines	0.76	10.30	1.56	19.48	1.19	11.73	6.1	7.65	Included with Paper
Fines	2.61	43.07	4.98	25.46	6.39	32.71	9.1	35.50	6.6
Total Weight of Grab, in lbs	784.7 - 29.94		642.25 - 26.81		544.2 - 36.59		303.0 - 23.59		

(*) Average of Kaiser 1968, 1972, and Drobny 1971 data, normalized.

(†) Percent loss on heating at 100 C.

Note: All weighings were read to the nearest 1/4 pound.

TABLE A-2. ANALYSIS OF REMIXED AND GROUND REFUSE FROM GRAB SAMPLE

Analysis As Indicated	Run 16		Run 17		Run 18		Run 19/20		Remarks
	4 mm	0.5mm	4 mm	0.5mm	4 mm	0.5 mm	4 mm	0.5 mm	
% Moisture	29.94		26.81		36.59		23.59		L-0-100 C
% Ash	15.10	16.37	10.24	11.69	7.62	-	5.86	6.05	LOI (960 C)
% Ash		16.2		12.1		10.0		6.2	LOI (500 C)
% Si in Ash		10-20		10-20		10-20		10-20	OES
% Ca in Ash		5-10		5-10		10-20		5-10	OES
% Na in Ash		2-4		5-10		3-6		3-6	OES
% K in Ash		2-4		2-4		3-6		3-6	OES
% Al in Ash		3-6		5-10		3-6		10-20	OES
% Mg in Ash		1-2		2-4		2-4		1-2	OES
% Fe in Ash		3-5		3-5		1-2		1-2	OES
% Ti in Ash		2.0		3.0		1.0		1-2	OES
% Pb in Ash		0.3		0.4		0.2		0.4	OES
% Zn in Ash		0.4		0.2		0.1		0.3	OES
% Cu in Ash		0.2		0.2		0.2		0.2	OES
% Mn in Ash		0.2		0.1		0.1		0.05	OES
% Ba in Ash		0.3		0.1		0.3		0.1	OES
% Cr in Ash		0.04		0.04		0.04		0.07	OES
% Zr in Ash		0.02		0.04		0.01		0.01	OES
% Sn in Ash		0.02		0.02		0.01		0.01	OES
% Ni in Ash		0.005		0.02		0.005		0.005	OES
% Mo in Ash		0.005		0.005		0.005		0.007	OES
% Cl in Ash	0.011	0.006	0.020	0.018	---	---			Solid Waste Laboratory
% Loss on ignition	84.90	83.63	89.76	88.31	92.38	---	94.14	93.95	Solid Waste Laboratory
% C in dry sample	42.92	41.82	45.06	42.94	48.19	---	47.24	45.93	Solid Waste Laboratory
% H "	5.60	5.46	6.13	5.76	6.47	---	6.37	6.14	Solid Waste Laboratory
% N "	0.57	0.78	0.54	0.56	0.91	---	0.75(*)		Solid Waste Laboratory
% Cl "	0.54	0.44	0.79	0.70	0.32	0.36	0.37	0.38	Solid Waste Laboratory
% S "	0.20		0.20		0.20		0.20		Based upon other refuse(†)
%00 "	35.1	34.9	37.0	38.2	36.28	---	39.21	40.55	By difference
Btu/lb	7691	7548	8083	7742	8886	---	8523	8164	

(*) Estimated on basis of average for similar samples.

(†) Twenty-one other samples gave S contents of 0.10 to 0.37 percent, averaging 0.19 percent.